

## 4 MAJOR PLANS FOR THE FUTURE

### 4.1 Plans for Future Instrumentation and Technique Development

#### 4.1.1 X-ray Imaging and Microscopy

In the future, the x-ray imaging and microscopy program will shift from serving primarily as a development and proving ground for strategic instrumentation and techniques to a more balanced program with greater emphasis on scientific applications. This shift is consistent with completion of the planned technical capabilities of the SRI-CAT beamlines and experimental stations. One area to be scaled back is the microfabrication and lithography effort, which will be taken over by the Center for Nanoscale Materials (CNM, see below). The development program will focus on three technical areas: soft and hard x-ray focusing optics with spot sizes down to the nanoscale; high-resolution imaging, diffraction, and coherent scattering using these optics; and high-throughput tomography. X-ray nano-probe development and nanoscience applications, especially in the soft x-ray region, will be conducted in support of the CNM to complement hard x-ray experiments at the proposed NanoCAT. Other applications of these techniques and instrumentation will emphasize investigation of outstanding problems in the biological, biomedical, environmental, and materials sciences. Examples include trace-elemental analysis by microfluorescence of phagosomes infected by pathogenic mycobacteria, and study of layer dynamics in liquid-crystal gels near phase transitions by x-ray intensity fluctuation spectroscopy.

#### 4.1.2 High-Energy X-ray Scattering

The SRI-CAT high-energy program will continue to pursue research along two complementary lines: high-energy x-ray optics development and high-energy experimental technique development. The optics portion of the program will continue to develop state-of-the-art high-energy x-ray optics, including focusing and narrow energy bandpass monochromators, micro-focusing with zone plates, and the use of multilayers and refractive lenses. The experimental-techniques portion of the program will emphasize the areas of high-energy experimentation that exploit the high-brilliance, high-energy x-rays provided by undulators at the APS. Brilliance is used by experiments that need small beams (e.g., 3D stress or compositional profiling), or by those needing very good angular resolution (e.g., high-resolution powder diffraction). Additionally, we will emphasize the development of *in situ* measurements and the study of transient behavior in materials. In the long term, the future of the high-energy x-ray program is strongly coupled with the proposed HEX-CAT. If the HEX-CAT beamlines are built, they will have a strong scientific focus and most of the programmatic science will be moved from 1-ID to HEX-CAT. The high-energy program will continue to use 1-ID for optics and other instrumentation development, consistent with the SRI-CAT mission.

#### 4.1.3 High-Resolution X-ray Scattering

The scientific programs for inelastic nuclear resonant scattering will be extended to

liquids in addition to current high-pressure studies exceeding 1 Mbar, thin films and surfaces and dynamics of proteins. The studies with Fe and Sn will be extended to Dy, Sm, Eu and Kr, as a regular part of the user program at 3-ID. The momentum-resolved inelastic x-ray scattering spectrometer will be enhanced with multiple analyzers. The overall performance of both programs will be improved by implementing cryogenically cooled, artificially linked monochromators. A new initiative will be evaluated in x-ray metrology for high-precision determination of x-ray wavelengths, lattice constant measurements, and x-ray interferometry, in particular with Michelson-Morley and Fabry-Perot type interferometers, taking advantage of exact backscattering or normal incidence diffraction beamline at sector 1.

#### 4.1.4 Polarization Techniques

Magnetic x-ray diffraction, reflectivity, diffuse scattering, and spectromicroscopy techniques will be extended to the study of nanoscale magnetic structures, spin-polarized electronic excitations and organic magnets. Of particular interest is the characterization of periodic magnetic arrays. Modern fabrication techniques have reduced the size of individual elements to as small as 40 nm, and our goal is to achieve a comparable spatial resolution with the circularly polarized microprobe. An upgrade of the existing photoemission microscope (PEEM) is planned in order to study structures down to 20 nm. Furthermore, we will work towards acquiring high magnetic field ( $B > 5$  T) capabilities on both branch lines to fully exploit the study of hard and dilute magnetic systems with polarized radiation. In addition, by employing

resonant coherent x-ray beams at L-edges of magnetic transition metal and rare-earth atoms the study of magnetic speckle and magnetic domain dynamics will be explored.

#### 4.1.5 Time-Resolved Program

Future efforts of the time-resolved program will fall into two categories: studies that utilize the pulsed nature of the storage ring and those that do not. In the first case, development of a fast x-ray streak camera will allow the development of techniques, such as image-correlation spectroscopy for pump-probe experiments. These will serve as prototype experiments for the kinds of experiments planned for the x-ray FEL sources when they come on-line. For the second case, emphasis will be given to the continued study of transient material behavior and of the dynamics associated with slow fluctuations in condensed matter. Coherent x-ray beams will be used to probe slow dynamics in condensed matter systems through the detection of speckle and the application of photon intensity correlation spectroscopy. This technique should lead to wide application in studying surface fluctuations in various solids and liquid films. In addition, using the technique of x-ray fluorescence correlation spectroscopy demonstrated by us on sector 2, we plan to study the dynamics and relaxation of nanoparticles inserted in complex fluids, interdiffusion at interfaces, and related phenomena. In many cases, these projects will be strongly coupled to the high-energy program or sector 1 optics programs.

## 4.2 User Support

### 4.2.1 Synchrotron Radiation Instrumentation Engineering

Engineering and technical support for users will continue to be the priority for both divisions. The main emphasis of this effort will be to develop beamline components and x-ray instrumentation for SRI CAT and other CATs. New front ends capable of handling the thermal loads of double-undulator straight sections will be developed. Users will be assisted with thermal, vibration, and beam stabilization studies, with nanomotion control, and with imaging software development. Some R&D in the area of magnetic systems, vacuum techniques, and software will be required to maintain state-of-the-art capabilities.

### 4.2.2 Insertion Devices

A program of occasionally swapping out insertion devices will be instituted to allow for improvements in the magnetic tuning of IDs that have been installed for a long time. The ID magnetic field will then be refined to meet the more stringent requirements of smaller storage ring emittance, and the devices will be checked for any magnetic degradation. Undulators with new period lengths will be designed and built as requested by users. The engineering of new, state-of-the-art insertion devices, such as the CPU, will continue and novel short-period superconducting devices will be developed. Effort will be directed towards providing a solid experimental basis for engineering the next-generation synchrotron facility through the engineering and operations support of the APS FEL (LEUTL) and the continuing collaboration with the LCLS.

### 4.2.3 Optics Fabrication and Metrology

New or enhanced x-ray optics fabrication and characterization facilities in support of new initiatives have been under development. The demand from the synchrotron user community has been for more control over reflecting and diffracting surfaces and interfaces. These demands are expected to increase with the arrival of x-ray FEL beamlines. Precise measurement of the power spectrum for surfaces at long length scales is accomplished with a long trace profiler, and we have added a second LTP instrument to our facility. This unit is currently set up and will serve as a test bed for LTP improvements. The power at short length scales will be measured with an atomic force microscope, and the addition of a near-field scanning optical microscope is planned. Diffuse x-ray scattering is an excellent tool to measure the power spectrum, and a program to implement such measurements on surfaces and interfaces of importance to APS users has been under active development. This program addresses the perpetual issue of the meaningfulness of non-x-ray-based metrology tools for the measurement of the performance of x-ray optics. Fabrication facilities for improved polishing have been built up and will be enhanced incrementally. Commercial suppliers of superpolished surfaces have been able to supply “roughnesses” at the sub-0.1-nm level (rms).

We will be in position to not only fabricate, but also to characterize, such surfaces over the full power spectrum and to verify these assessments with x-ray measurements. The deposition facility continues to be improved and enhanced in several areas. Control over

uniformity for long deposition runs has been a focused activity. These efforts are important for achievement of high-reflectivity multilayers and supermirrors. Design and use of sputtering masks will continue to be emphasized for the growth of laterally graded multilayers and for making the next-generation Kirkpatrick-Baez mirrors using the differential deposition technique. Finally, we will exploit two capabilities that have recently been added. A dicing saw has been commissioned for nanofabrication purposes and for making x-ray analyzers. This saw has already seen significant use for these purposes. A CCD camera has been added to the rotating anode facility for the purpose of making quantitative assessment of topography data and to facilitate the collection of diffraction data. A program to evaluate stress in thin films and multilayers will be undertaken with the addition of this new CCD. Assessment of multilayer performance at cryogenic temperatures is planned.

#### 4.2.4 High-Heat-Load Optics

The high-heat-load optics program has been focused for several years on solving the problem of developing monochromators to handle the heat load from APS undulators. This goal has been met, and the work on high-heat-load monochromators has subsided to some degree. We still have several high-heat-load projects (refinement of monochromator designs, multilayer monochromators, high-energy monochromators) on which we are working, but a substantial part of our research efforts is now being directed to other optical related areas, such as x-ray interferometry and phase-contrast imaging.

### 4.3 New Initiatives

#### 4.3.1 Center for Nanoscale Materials

The Center for Nanoscale Materials is one of the five nanoscience research centers proposed by the Department of Energy (DOE). This center builds on strengths at ANL in nanosciences and hard x-ray microprobes to create a unique national capability for nanoscale synthesis, fabrication, and analysis of materials. The CNM will explore the behavior of materials confined to length scales smaller than those that define macroscopic physical behavior. In addition, it will draw extensively on a wide range of resources at ANL, especially the APS, to create an organization at the forefront of nanoscale materials research. The CNM will include an approx. 40,000 sq. ft. building attached to the APS on the southwest side of the Experiment Hall. The CNM will include the development of a new sector at the APS and establishment of NanoCAT. The aim of NanoCAT is to apply a multitude of x-ray techniques to the study and development of advanced nanostructured materials and nanoscale devices at the CNM. The efforts within the proposed NanoCAT will be integrated with the design, characterization, and fabrication efforts within the CNM. In addition, beamlines in the existing SRI CAT will work cooperatively with the CNM to carry out upgrades and subsequent experimental programs in support of the research activities at the CNM.

The XFD will be providing effort for the conceptual design of the NanoCAT sector, including a hard x-ray nanoprobe ID beamline, an x-ray scattering ID beamline

for *in situ* real-time analysis of nanomaterial processing, an x-ray scattering BM beamline for rapid turn-around analysis of nanostructures in support of nanofabrication, and an x-ray lithography BM beamline. XFD scientists are working with scientists from MSD and the ANL Chemistry Division to provide the project planning for the CNM and R&D in support of construction for both synchrotron radiation techniques and microfabrication techniques critical to its success. During construction, XFD will provide scientific and engineering support in the completion of the NanoCAT sector and related facilities at the CNM. XFD scientists will continue to develop experimental techniques and conduct preliminary experiments that will contribute to the basis of the x-ray science that will be part of the CNM.

#### **4.3.2 Inelastic X-ray Scattering (IXS) CAT**

A dedicated inelastic x-ray scattering beamline at the APS is proposed, building on the experience obtained at sector 3 of SRI CAT and at other third-generation sources. This proposal, submitted to DOE and the National Science Foundation by a group of scientists at national laboratories, universities and industrial research laboratories, would deliver a state-of-the-art beamline in two complementary energy regimes and would attack a broad scientific program. One spectrometer will focus on very high-resolution work, with energy resolutions in the sub-meV regime. This spectrometer will have the highest resolution in the world and significant projected flux increases of the order of 10. It will carry out seminal work in the study of dynamics in systems, such as high-temperature superconductors, solids

under high pressure, proteins and polymers. The second spectrometer will be optimized for the study of higher energy excitations, with resolutions in the 0.05-1 eV range. Here studies will be carried out on electronic excitations in electronically active materials, such as the colossal magnetoresistance manganites, high-temperature superconductors, semiconductors and other novel materials, including the  $C_{60}$ -based conductors and Mott-Hubbard insulators.

These two components of the CAT will be designed and operated as essentially separate beamlines, with independently optimized undulators, monochromators and spectrometers. It will take advantage of the next-generation short-period and/or superconducting undulators. In a certain sense, the IXS-CAT beamline may be said to be the "first second-generation beamline at a third-generation source" to constitute the premier facility for the study of dynamics with x-rays in this country.

#### **4.3.3 Normal Incidence Diffraction Beamline at Sector 1**

The possibility of building a normal incidence diffraction beamline at the APS was considered early, during the construction phase of the APS, and provisions were made in the storage ring magnets in sector 1 and the storage ring tunnel to extract the photons into the "Early Assembly Area" where there are no other beamlines. In 2000, efforts were made to observe the beam and to characterize the reflectivity and energy bandpass of the back-reflected beam. The encouraging results obtained with sapphire and silicon crystals provided the possibility to generate high-energy-resolution x-rays with a bandpass of

1-to-100 meV in the energy range of 4-to-40 keV. The tunability of up to 20 eV at many different energy points is feasible, with the additional possibility of matching absorption edges of elements. The photon flux exceeding  $10^{13}$  ph/sec/100 meV at 6 keV, or  $10^{11}$  ph/sec/10 meV at 15 keV, or even  $10^{10}$  ph/sec/1 meV at 25 keV can be reached. The total length of the beamline can exceed 130 m, if the backscattering crystal is placed at the end of sector 1.

There are a variety of scientific possibilities offered by a normal incidence diffraction beamline: x-ray metrology, x-ray interferometry, high-energy resolution spectroscopy above 30 keV, and microfocusing for a submicron-resolution Mossbauer microscope. We plan to pursue all of these possibilities.

#### 4.3.4 High-Energy X-ray (HEX) CAT

HEX-CAT is an effort to form a collaborative access team for the construction and operation of an APS sector dedicated to the use of high-energy x-rays for a diverse scientific agenda. HEX-CAT has primarily grown out of the high-energy x-ray program based on sector 1, and the CAT director and several members of the CAT are from the x-ray physics group of UPD. If HEX-CAT is funded and built, some members of the x-ray physics group will become essentially full-time HEX-CAT staff members. A letter of intent was submitted to the APS Program Evaluation Board in October of 2000. This letter of intent was accepted, and the Board invited HEX-CAT to submit a scientific proposal.

The ID beamline of the HEX-CAT will be a "second-generation" APS beamline, with specially designed undulators, state-of-the-art x-ray optics, and dedicated experimental instrumentation. The two primary goals of the beamline will be to maximize the x-ray brilliance in the 40 keV to 90 keV energy range and to have efficient operations to accommodate large numbers of experiments. An optimized high-energy BM beamline will also be built to provide for experiments that do not require the high levels of brilliance of the undulator beamline.

The scientific scope for the CAT is the use of high-energy x-rays for single-crystal and powder diffraction, diffuse scattering, scattering from amorphous materials, determination of stress/strain and texture in materials, phase-contrast imaging, fluorescence mapping and the study of solid/liquid or liquid/liquid interfaces. Dedicated instrumentation will be developed for each of these techniques. The intention of HEX-CAT is to serve a wide range of scientific interests including (but not limited to): condensed-matter physics, both applied and basic materials science research, chemistry, environmental and geological sciences, engineering, and nontraditional synchrotron users such as archaeologists. We believe that this facility will be highly complementary to other APS CATs and to the users of neutron scattering facilities.